

## Flotation of Sulphide Ores - HZL Experience

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### ABSTRACT

*Flotation process, patented in the year 1906, was originally developed for mineral industry to recover values from high grade tailings of gravity separation plants. This technology has acclaimed importance as a versatile process for the beneficiation of vast variety of sulphide minerals. Due to flexibility of the process and remarkable development taken place in flotation technology and its ancillary systems, it has now become possible to recover fine grained sulphide minerals from complex ore deposits, whose processing was earlier considered uneconomical. Today, about 400 million tonnes of sulphide ore is treated annually by flotation process worldwide.*

*M/s Hindustan Zinc Ltd. (HZL), a leading lead-zinc mining and smelting company in India has experienced a spectacular growth from the time of its inception. Starting from a single 500 MT per day lead-zinc mine at Zawar, HZL has expanded its mining capacity to about 13,000 MT per day with matching ore beneficiation plants, all adopting froth flotation technology.*

*The phenomenal progress is a result of HZL's emphasis on research and development with an objective to improve the efficiency and to optimize performance, and to seek innovative modern technology and control systems in their flotation plant and processes. This has not only solved metallurgical problems but improved overall plant economics. High grade concentrates produced with reduced deleterious impurities have further enhanced smelting efficiency.*

*The paper covers general principles and aspects of industrial sulphide ore flotation practices in brief and processes adopted in HZL's plants. It also highlights R&D efforts made and new technologies adopted in one of the most complex sulphide ore beneficiation plants to solve metallurgical problems in flotation process.*

**Key Words :** Froth flotation, Sulphide flotation, Control system, Flotation chemicals, Hindustan zinc, Ore beneficiation,

## INTRODUCTION

History of Sulphide ore flotation dates back to beginning of 20th century. Sulphide ores are major sources of base metals like Copper, Lead, Zinc, Nickel, Cobalt etc. Beneficiation of these base metal sulphide minerals is predominating through froth flotation technique. This technology has acclaimed importance as a versatile process for beneficiation of vast variety of sulphide minerals. Due to flexibility of the process and remarkable development taken place in flotation technology and its ancillary systems, it has now become possible to recover fine grained sulphide minerals from complex ore deposits, whose processing was earlier considered un-economical. Today, about 400 million tons of sulphide ore is treated annually by flotation process worldwide. General principles and factors effecting flotation process, control systems, innovations and new trends are reviewed in the paper.

M/s Hindustan Zinc Ltd, a leading mining and smelting company in India treats annually 3.5 million tons sulphide ore. Plant practise, R&D efforts made, new technologies adopted in one of the most complex sulphide ore beneficiation plants at Rajpura-Dariba mine highlight various practical aspects of sulphide ore flotation technologies.

## DISTINGUISHED FEATURES OF SULPHIDE MINERALS

Some of the important sulphide minerals are: Galena (PbS), Sphalerite (ZnS), Chalcocite ( $\text{Cu}_2\text{S}$ ), Covellite (CuS), Chalcopyrite ( $\text{CuFeS}_2$ ), Pyrite ( $\text{FeS}_2$ ), Molybdenite ( $\text{MoS}$ ) etc. Distinguished features of these minerals are:-

1. Above mentioned important minerals are covalently bonded compounds and possess very low solubility, each of these has a definite solubility product in water.
2. Sulphide minerals are meta stable and are prone to surface oxidation in presence of water and oxygen.
3. These are electronic semi-conductors, can act as source or sink for electron and therefore can support electrode reaction at surface.
4. Most of the sulphide minerals are regularly charged in working range of pH and therefore the electrostatic or electrical double layer interaction oppose the absorption of commonly used anionic reagents.

5. Some of the sulphide minerals have feeble natural hydrophobicity. Some of these are capable of rapid and complete flotation in absence of conventional collectors.
6. One of the important consequences of the electrochemical nature of the surface reaction is the galvanic interaction between the grinding media and the sulphide minerals.
7. Sulphide ore minerals are generally floated using thiol type reagent xanthates, dithio phosphates etc. Oxidised minerals do not respond to these collectors and hence require surface modifying treatment.

### **COMPLEX SULPHIDE ORES**

Many of the sulphide minerals do not occurs singly. They are generally associated with other sulphides e.g deposits of galena will have sphalerite, pyrite as major sulphide and chalcopyrite, pyrrhotite as minor sulphide minerals and at times association of precious mineral of gold and silver also. Some oxide minerals may be associated with sulphide minerals, particularly in upper zone of a deposit. Beneficiation of ores containing multi metals minerals to produce specified quality of separate concentrates is difficult. The problem becomes more challenging when such ore contain impurities which are difficult to remove and their presence in concentrates are not acceptable to smelters and heavy penalties are levied if these are beyond limits. Some major factors adding to complexity of sulphide minerals are

1. Intricate mineralogy of various minerals in close association with each other and gangue minerals.
2. Very fine dissemination of ore minerals in matrix.
3. Occurrence of oxide and oxidised minerals.
4. Excessive presence pyrite and pyrrhotite.
5. Presence of naturally floatable gangue minerals like talc, graphite, mica etc.
6. Occurrence of clays and other slimes interfacing in selectivity of separation.
7. Tarnishing of minerals surface after mining or during storage and transportation.

Processing of such complex sulphide ore deposit is difficult and challenging. Development of flow-sheet needs extensive laboratory investigations and pilot scale testing, such flow-sheet comprise many

stages of grinding and flotation, special treatment. It may also require integration of different beneficiation techniques besides conventional flotation.

## FLOTATION PROCESS

Flotation is a method of separating an ore species from another, based on its hydrophobic surface characteristics either natural or induced when present as a suspension in water with air bubble. Due to the affinity of the desired mineral to adhere to air bubbles it is floated out of the ore slurry. Schematics of various sub processes controlling flotation system is given in Fig.1.

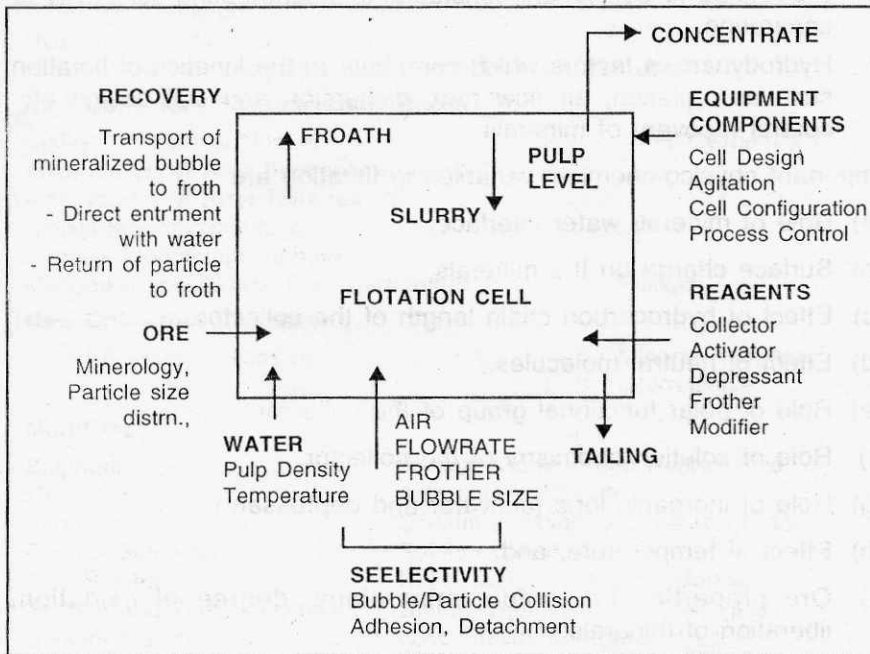


Fig. 1 : Various sub-processes controlling flotation system

The successful industrial practice of flotation involves knowledge and optimisation of four important components of flotation process namely,

1. Mineralogical characteristics of the ore (mineral association, liberation size, presence of slime particles and soluble species contributed by the ore).

2. Surface colloid and reagent chemistry which determines selectivity of separation (collectors, frothers, activators, depressant, modifier, dispersants etc.)
3. Process engineering (feed preparation that is size reduction , cell design, control system etc..
4. Operating parameters such as aeration rate, temperature, Eh/ pH, ionic strength and flotation circuit configuration.

Overall separation efficiency in flotation is dependent on

1. Surface chemistry factors such as particle bubble attachment, mineral reagent interactions, reagent chemistry etc. These factors are related to equilibrium considerations contributing selectivity to separation.
2. Hydrodynamics factors which contribute to the kinetics of flotation such as agitation, air flow rate, dispersion and cell design etc. control recovery of minerals.

Important physico-chemical variables in flotation are :

- (a) Role of mineral/ water interface.
- (b) Surface charge on the minerals.
- (c) Effect of hydrocarbon chain length of the collector.
- (d) Effect of neutral molecules.
- (e) Role of polar functional group of the collector.
- (f) Role of solution chemistry of the collector.
- (g) Role of inorganic ions (activator and depressant).
- (h) Effect of temperature, and
- (i) Ore properties i.e. grade, minerology, degree of oxidation, liberation of minerals.

## REAGENTS USED IN SULPHIDE FLOTATION

Commonly used reagents in industrial flotation plants are Collector, Frother, Activator, Depressants, pH modifier. Amongst above, collectors are the most important reagents which play critical role in sulphide flotation. Sulphahydryl or thiol type collectors which in general consist of "SH" group in combination with an organic radical have extensive application. List of collectors industrially used for sulphide flotation is given in Table 1

*Table 1: Traditionally used and new sulfide and modifiers  
(After Nagaraj, 1994)*

### **Traditionally Used Collectors**

Xanthates

Dialkyl and Dicresyl Dithiophosphates

Dialkyl Thionocarbamates

Mercaptobenzothiazole

Xanthogen Formates

Xanthate Ester

Dodecyl Mercaptan

Dialkyl Dithiocarbamate

Diphenyl Thiourea

Diphenyl Guanidine

Hydrocarbon oils

(Several blends comprising above collectors are also used)

### **New Collectors (Commercially Used)**

Dialkyl Dithiophosphinates

Cytec/Cyanamid

Alkoxy carbonyl Alkyl thionocarbarmates

„

Alkoxy carbonyl Alkyl Thiourea

„

Dialkyl Monothiophosphate

„

Dicresyl Monothiophosphate

„

Monoalkyl and Dialkyl Trithiocarbonates

Phillips

### **New Collectors (Pipeline)**

F and S series collectors

Mineral Reagents  
International

### **Modifiers**

Sulphuric acid

Sodium sulphide and Hydrosulfide

Lime

Ammonium sulphide

Soda Ash

Sodium sulphide and metabisulfide.

Copper Sulphate

Sulphur dioxide.

Zinc Sulphate

Nokes reagent, Arsenic Nokes.

Sodium and Zinc Cyanide

Starch, Guar gum, modified guar.

Sodium Silicate

CMC, dextrine, organic dyes.

Legnin Sulfonates

Sodium thioglycolate, Mercaptoethanol  
and trithiocarbonate derivatives of above.

**Discontinued or Very Limited Usage : Chromate, Ferro and Ferri Cyanide,  
Quebracho, Permanganate**

Extensive research has been carried out to understand surface chemistry and interaction between collector and sulphide. It is not possible here to deal with the theories as various phenomena are



observed in actual flotation system are yet to be fully investigated. Some of the important aspects are listed below :

1. Interaction between sulphide minerals and thiol collectors takes place by a corrosion type mixed potential mechanism involving simultaneous electrochemical reactions at mineral water interface.
2. Potential difference of the mineral solution interface is the most important parameter determining the rates of reactions causing flotation which is termed as 'Redox potential' (Eh). By monitoring oxidation/reduction environment of the pulp, (Eh) can be controlled which can be used as one of the on-line control parameters of flotation process in the plant for naturally or feebly floatable sulphide minerals.
3. In order to enhance the selectivity of collection many modifying agents like activator, depressor are used for soluble sulphide minerals.

However flotation kinetics/selectivity of many soluble sulphide eg. sphalerite and pyrotite requires activation under normal flotation conditions. Longer chained xanthates are generally used for improving selectivity; research work has confirmed relationship between carbon number, flotation response and solubility product of corresponding metal xanthates. Xanthate collectors are not capable of recovering ultra fine particles i.e  $<10$  and significant losses can occur in the fine fraction. They are not selective with respect to iron sulphide. The average consumption of xanthate is relatively very high ranging from 50 to 150 gm/MT. Therefore mixture of two / three types of xanthates at different stages of collection also in combination of dithiophosphates and certain nitril compounds are being practised in many sulphide flotation plants.

Another important parameter is pH, which has remarkable effect on the flotation of sulphide minerals. It is established that xanthates decompose in acidic environment and hence, results lower flotation recovery. Through proper control of pulp pH, one sulphide can be selectively floated from other.

As regards other reagents used in sulphide flotation, depressants like sodium cyanide,  $\text{ZnSO}_4$ , sodium meta bi-sulphite are common. Effectiveness of depressant also depends on concentration and selection of collectors. In case of cyanide as iron sulphide depressant there is a relationship between concentration of collector and cyanide ion present. At low pH higher quantities of cyanide is required. In this

case, pyrite ferrocyanide complex formed is also responsible for depression, combination of  $\text{ZnSO}_4$  and  $\text{NaCN}$  is also common.

Various sulfoxo compounds (e.g alkyl sulphates) are also used for depression of sphalerite. Cyanide dissolves surface copper from activated sphalerite and can react with iron and zinc xanthate to form soluble complexes, eliminating xanthates from the surfaces of the minerals of these metals. Soda ash is used to regulate pH if pyrite is very high.

For depression of galena in Pb-Cu separation potassium dichromate,  $\text{SO}_2$  are commercially used. Thermal treatment i.e rise in pulp temperature ( $700^\circ\text{C}$ ) has been successfully tried. Nitrogen and carbon dioxide gases are also used for galena depression. Galena flotation is preferred at lower pH than sphalerite. Lime is used to control pH and to depress pyrite but it can depress galena to some extent.

For activation of sphalerite, use of copper sulphate is universally practised. Prior to activation of sphalerite with  $\text{CuSO}_4$  proper conditioning to maintain alkaline pH is necessary. This prevents pyrite activation.

Pine oil, M.I.B.C and cresylic acid are commercially used frothers in sulphide flotation depending upon overall economics and nature of frother whether brittle or hard, and froth bubble size required.

If oxides and oxide mineral are associated then sulphidising by sodium sulphide is widely adopted followed by flotation with xanthate or metacaptobenzols and mercaptans. Sulphidising process need to be monitored carefully, generally done in stages and preferably through control of pulp Eh. Chelating agents and use of cationic collectors for flotation of oxidised minerals are also practised.

Heavy metal ions are present in slurry water if ore is slightly oxidised. Sphalerite can become activated by heavy metal ions in solution. Lime and soda ash is used to precipitate these heavy metal ions.

## **INNOVATIONS IN FLOTATION**

In view of increasing mineralogical complexity and depleting grade and general recession in mining industry during seventies and eighties, it became necessary to seek improvement in mineral processing technology. Priority was to reduce operating cost, particularly energy cost and development of new technique to improve metal recovery and plant throughputs. Automation in mineral processing plants become essential.



Developments taken place in flotation technology are in following major areas :

1. Increase in size of flotation cells with better energy efficient design.
2. Development of speciality reagents for particular application or specific type of ore.
3. Research oriented towards better understanding of surface chemistry and micro processes involved and kinetics of flotation with respect to variables.
4. On-line chemical analysis, automatic process control and computer applications.

### **Large Size Flotation Cells**

In order to improve capital and operating costs per unit of metal produced, large size mining and mineral processing equipment's were developed. In case of flotation cells, in the modern plants cell size of 43 M<sup>3</sup> is quite common. Advantages of large size flotation cells claimed are saving in floor space, low power consumption, low wear rate, high selectivity, reduction in reagent consumption and easy start up on full load.

Flotation columns developed in sixties for phosphate and coal flotation, gained popularity during eighties in sulphide ore flotation of copper and molybdenum, particularly in cleaning and recleaning stages. One single flotation column can replace a bank of mechanical flotation cell. Flotation columns are now extensively installed in lead, zinc, molybdenum, gold sulphide ore flotation plants. Potential benefits of flotation columns are high capacity at less floor area, no moving part so less wear, very high grade of conc., less entrainment of gangue due to froth washing better fine particle recovery, better control at low cost.

Flotation column technology underwent many innovations mainly air sponger systems, instrumentation & controls, feeding system. Leeds column, Jameson cell, packed column technique, microcel column are new developments ore industrially adopted.

Other developments in sulphide ore flotation are Skim air/Flash flotation to recover liberated coarse particles in grinding circuit itself. Application of selective flocculation in flotation to recover ultrafine desired mineral or to reject gangue minerals.

### **Research in Development of Flotation Chemicals**

Xanthates continues to dominate sulphide ore flotation industry whereas initial usage of dithiophosphates and thionocarbonates were coupled with xanthate collectors, these one now gaining demand. Most of these flotation chemicals used today were being produced and industrially available by 1960. Serious research and development started in late seventies due to recessing in mining/metal industry. Due to complexity of beneficiation problems associated with lean and difficult (to float) ores, it became imperative to develop more selective, almost tailor made reagents for specific applications. The major areas are :

- (a). Reagents with reduced dependence on pH control for their effectiveness.
- (b). Better selectivity over pyrite, pyrrhotite, talc, graphite which are problematic in most of the sulphide flotation.
- (c). Improve recovery of relatively coarse and partially liberated particles.
- (d). Enhance recovery of precious metals and other minor metal of strategic importance.
- (e). More effective differential flotation amongst sulphide minerals (Cu/Mo, Cu/Zn, Pb/Zn, Cu/Ni/Co etc.).
- (f). Better recovery of oxidised sulphide minerals.
- (g). To check entrapment/entertainment of fine gangue during flotation.
- (h). Development of cheaper reagents and substitution of environmentally hazardous in-organic reagents like cyanides, dichromates, and development of bio-degradable reagents to replace organic reagents.

### **CONTROL SYSTEMS IN FLOTATION CIRCUITS**

Economics of a beneficiation plant depends on performance of flotation circuits i.e grade of concentrate and recovery. Flotation process is a complex process involving many variable and parameters, many of their fundamental mechanisms which determine the efficiency of the process are not understood, some parameters are not easily measured, can result in slow and erratic response of the circuit to control system.

Some major source of disturbances are un-uniform feed, ore characteristics, grind size, feed flow, pulp density and in addition sudden build up of circulating loads and spillage due to malfunctioning of ancillary equipment. In the past flotation operators have been able to achieve high process efficiency, by visual observations to characterizes circuit performance. For improving concentrator economics it is essential to exercise effective control reliable measurement and control equipment are essential for quick detection of disturbances and to take counter action immediately.

### **On-line Chemical Analysis**

It is of foremost importance in any ore beneficiation plant to know continuous trends of assays at critical stages i.e. feed, concentrates and tails. Number of in-stream, on-stream, near-stream analyses systems have been developed and working efficiently in many plants throughout the world. Some names are AMDEL, Australia. Boxray-Boliden Sweden, corrier- Outokumpu Finland.

### **Flotation Control Systems**

Flotation process variable to be monitored for control system:-

- (a) Reagents flow at various points.
- (b) Regulation of pH.
- (c) Bank pulp level control.
- (d) Regulation of bank aeration rates.

Before development of control system it is required to define the objectives considering overall economics and policy of the company. Well-desinged modern control system have an orderly hierarchical structure, three levels of controls in general are :

- (a) Basic or regulatory control which involves the control of a particular set point which may be either analogue or digital.
- (b) Stabilising control- involves calculation of the value of the set point at which the process variable to be operated using other process measurement. This controller compares the efficiency of the process against some performance criterion and from the difference determines the set point at which the process variable need to be operated.
- (c) Optimising control which determines optimum performance at which the process should be operated. This needs mathematical modeling and simulation.

These three levels of control may be linked in such a manner that the lower level may still operate even if higher levels do not. Some of the flotation control systems developed and working worldwide are :

J K Tech & MIM consortium, Bailey DCS of Australia, 'Procon', of Outokumpu Finland, T D C - 3000 of Honeywell - U.S.A, 'Floatstar' of Mintek South Africa, Control International S.A (BRGM) of France etc.

Recent trend is to develop expert systems using model based multivariable auto control system to take care of interaction of various single control loops and integration of operators practical knowledge and experience for optimisation of the process in a particular plant. Advanced model based multivariable auto control systems using very recent concepts like adaptive control, fuzzy logic, expert systems and neural networks to suit a particular beneficiation plant have also been effectively installed and operated throughout the world.

### **Some Practical Aspects of Control System**

It is advised to develop a control system after extensive metallurgical plant performance data collection and study of circuit configuration. Higher level control must be designed after stabilisation of automatic analysis system and basic regulatory control loops as ore properties and circuit constraints vary from plant to plant.

Control objectives are generally rationalised in terms of grade/ recovery curves as shown in Fig. 2. The aim of the control system should be to move the operating curve C, which will provide optimum metallurgical performance. The target operating point 'P' on the curve C (normally defined by specifying the required concentrate grade) can then be attained by manipulating necessary process variable.

Prior systematic plant auditing can reveal many of small but critical points which may be of great importance about various equipment capacities, constraints on circuit configuration for designing control system. For example, sources and reasons for circulating load fluctuations and closely related matters of concentrate production, mainly feed rate and feed grade. If rougher circuits are operated efficiently, control of succeeding stages become easier. It may not be appropriate to attempt for very low final tailing grade which may not be 'realistic' as it will result in very high flow of low grade scavenger concentrate lowering final concentrate grade-gangue contamination of concentrate can be minimised by measurement and automatic control of flotation cell levels and suitable aeration.

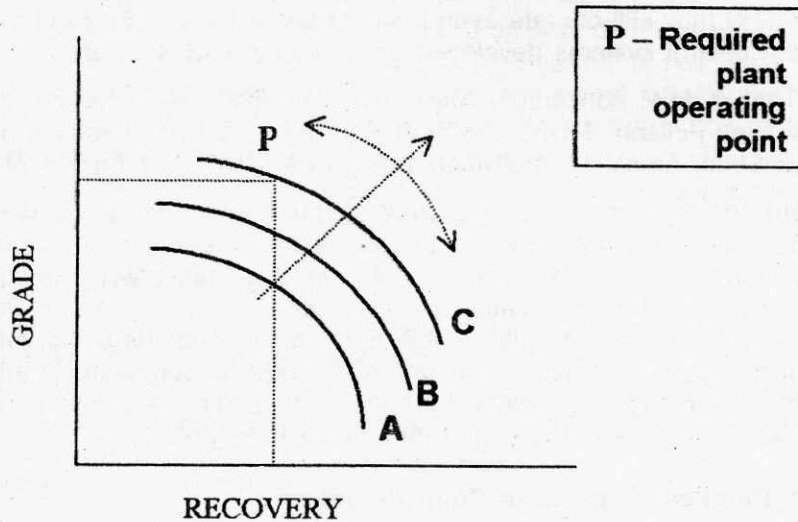


Fig. 2 : Typical grade-recovery curves

A single feed analysis system is sufficient if the feed slurry is splitted into number of flotation circuits. But tailing of individual circuit must be essentially analysed. For complex ore with fluctuating feed grade or varying undesired minerals like Pyrrhotite, resulting build up of circulating load it is desirable to measure/ control following elements:

1. Air flow rate to each bank or different sections rougher / scavenger/ cleaners.
2. Flow rates of key circulating load streams such as scavenger concentrate and cleaner tailing .
3. In stream analysis of key intermediate streams as rougher/ scavenger concentrates and cleaner tailings.

For reagent control, statistical relationship between reagent doses and assay at important flotation stages and including misplacement of metals should be worked out. Control of collector addition rate is often performed by feed-forward control based on a linear response to assay or tonnage of valuable metal in the flotation feed. Although feed-forward control can provide a degree of stability, stabilisation may be more effective using feed back data. Feed back loops utilising tailing

assay overcomes the distance - velocity lag experienced with feed back loops.

Another important aspects of effective control system is control of flotation feed characteristics i.e pulp density and size distribution. It is advisable to install essential instrumentation in grinding circuits auto control system is ideal but will depend on overall economic consideration.

Flotation circuits control should be implemented in stages :

- (a) Instalment of measuring instruments and basic regulatory control loops.
- (b) Development of operating practice to make full use of on-line data from these instruments.
- (c) Introduction of higher level control loops for optimisation.
- (d) Development of the control system through monitoring / calibration/ upgradation using practical skill and experience of plant engineers.

### **Economical Benefits**

The economic benefits which have been derived from flotation control system come from one or more of the followings :

- (a) Increased metal recovery.
- (b) Increased concentrate grade.
- (c) More uniform product .
- (d) Lower reagent consumption.
- (e) Lower manpower requirement.

### **HZL'S EXPERIENCE IN SULPHIDE ORE FLOTATION**

HZL's spectrum of activities ranges from exploration, mining and ore beneficiation to smelting and refining of lead, zinc, cadmium, silver, cobalt etc. HZL operates 5 lead-zinc mine with a total ore production capacity of 3.5 million tonnes per annum with matching ore beneficiation plants.

For recovery of galena and sphalerite minerals, differential flotation was adopted in the year 1950 at Zawar, still it continues in other plants including latest and most modern 4500 TPD plant at Rampura Agucha. Single stage grinding in ball mills (except rod mill- ball mill combination



at Rampura Agucha), regrinding of middling is also practised. Conventional process of galena flotation (after depressing sphalerite & pyrite with  $\text{ZnSO}_4$  and  $\text{NaCN}$ ) followed by activation of sphalerite with zinc sulphate and flotation at 9 to 10 pH. Potassium ethyl and sodium isopropyl xanthates are used as collector whereas MIBC and cresylic acid as frothers. While Zawar ore is easy to float and clean concentrates are produced, problem of graphite carbon (upto 12%) in lead concentrate of Rampura Agucha and Rajpura Dariba mine persists. Similarly, at Rajpura Dariba mine zinc concentrate contains high silica (up to 6%). Such impurities are not acceptable.

HZL's Smelters desired concentrate grade is zinc +52%, and Pb +60%. Zinc metal recovery in flotation is in the range of 85% to 90% in all major plants. But recovery of Pb varies from plant to plant. At Rampura-Agucha mine Pb. Concentrate grade is 50% at 50% recovery due to complex mineralogy. Silver is found in HZL ores (20 to 45 ppm), no special treatment for silver adopted, its recovery is about 75%.

HZL has built in-house R&D facilities to solve process problems and to adopt new technologies. At Central Research and Development Laboratory, Debari, a well equipped beneficiation laboratory including complete batch testing equipment and pilot plant of 2 TPD capacity has been established. To support ore beneficiation, well equipped petromineralogy laboratory for detailed ores mineralogical investigation of ores and beneficiation products has significant contribution. For development of beneficiation flowsheets and solving critical process problems in their own plants, consultancy of R&D institution in India and abroad in the field of ore beneficiation is sought as and when required. Some of the conventional flotation techniques adopted in-house are Cu-Pb bulk flotation and graphite flotation, unit flotation, flash flotation, pyrite flotation on plant scale. Various new speciality reagents to improve Pb-Zn and silver recovery, rejections of pyrite and gangue minerals have been extensively tested at plant scale.

HZL is pioneer in introduction of in-stream analyser, automatic control system and adaptation of new technologies in grinding and flotation circuits. This has improved performance of all the beneficiation plants. One such system is shown in Fig. 3.

More than three decades of plant operations, R&D work, installation and commissioning of new plants, introduction of new technologies have developed expertise in sulphide ore beneficiation process at Hindustan Zinc Ltd. Beneficiation process and developments at

Rajpura-Dariba concentrator described in the next section, illustrates this.

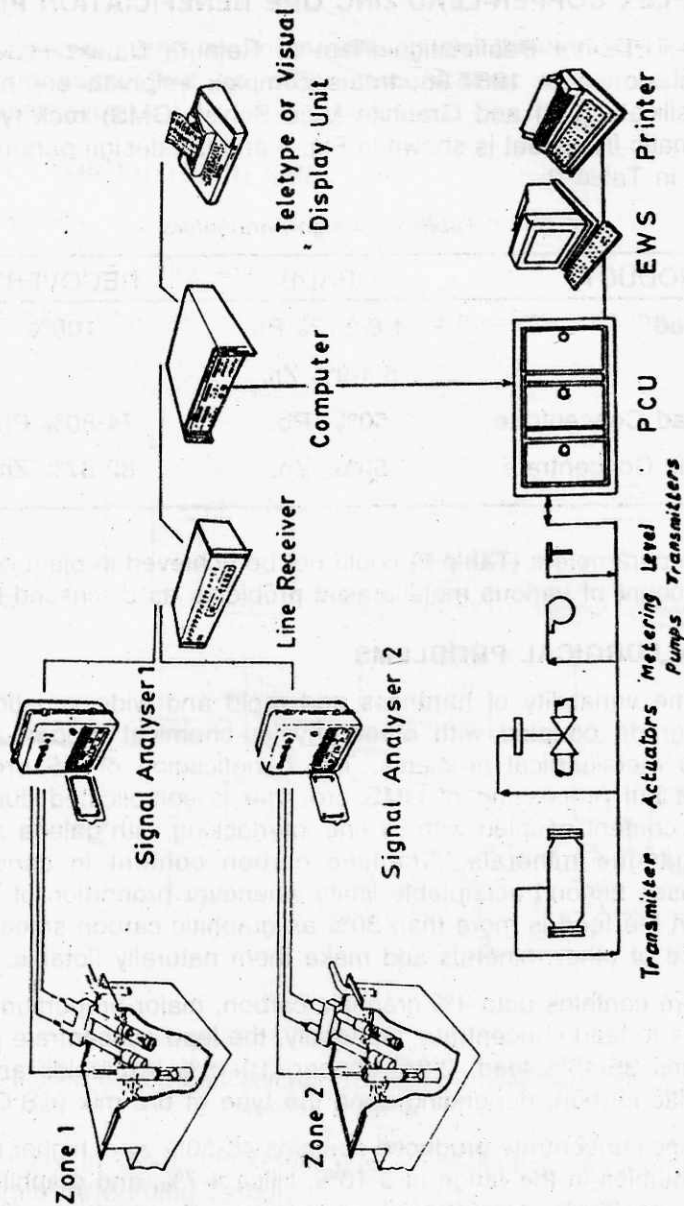


Fig. 3 : On-line analysis and automatic control system

## DEVELOPMENT WORK & NEW CONCEPTS ADOPTED IN HZL' S COMPLEX COPPER-LEAD-ZINC ORE BENEFICIATION PLANT

3,000 TPD Ore Beneficiation Plant at Rajpura Dariba Mine (RDM), commissioned in 1984-85, treats complex sulphide ore hosted by Calc-silicate (CS) and Graphite Mica Schist (GMS) rock types. The schematic flowsheet is shown in Fig. 4 and the design parameters are given in Table 2.

*Table 2: Design parameters*

PRODUCT	GRADE	RECOVERY
Feed	1.6-2.6% Pb 5.4-9% Zn.	100%
Lead Concentrate	50% Pb.	74-80% Pb.
Zinc Concentrate	53% Zn.	82-87% Zn.

These parameters (Table 2) could not be achieved in plant operations on account of various metallurgical problems as discussed below.

### METALLURGICAL PROBLEMS

Extreme variability of hardness and rapid and wide variation in mill feed grade coupled with other physico-chemical properties of ore create metallurgical problems. The beneficiation of CS ore type is simple but processing of GMS ore type is complicated due to high pyrite content coupled with its fine interlocking with galena sphalerite and gangue minerals. Graphitic carbon content in concentrates increases beyond acceptable limits whenever proportion of GMS ore type in the feed is more than 30% as graphitic carbon smears on the surface of other minerals and make them naturally floatable.

The ore contains upto 4% graphitic carbon, major proportion of which reports to lead concentrate. Generally, the lead concentrate produced contains 35-45% lead, 2-3% copper, 10-15% insolubles and 6-10% graphitic carbon, depending upon the type of ore mix (CS:GMS).

The zinc concentrate produced contains 46-50% zinc, higher incidence of insolubles in the range of 5-10%, silica 4-7%, and graphitic carbon 2-3% causing operational problems at smelting stage. The specifications of concentrates desired by smelters and the average

concentrate grades produced at RDM during the last 5 years are given in Table 3.

In stream analysis auto control in grinding and flotation circuits were installed in year 1992 for efficient control in view to improve metallurgical results.

## EFFECT OF IMPURITIES IN SMELTING

(a) High graphitic carbon in lead concentrate leading to

- Reduction in sinter strength,
- Difficulty in temperature control during roasting/sintering,

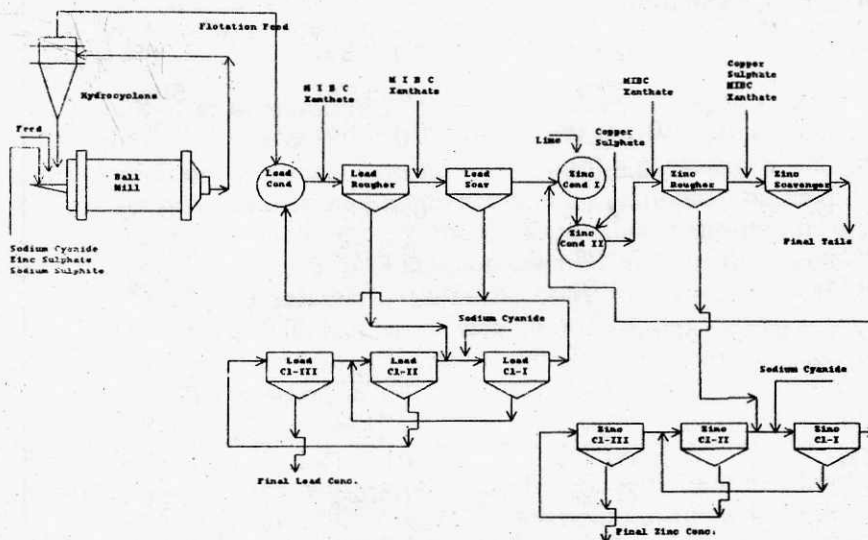


Fig-4 : Rajpura Dariba Concentrator : Schematic flowsheet with reagent addition scheme

- Difficulty in desulphurization due to excessive requirement of oxygen for burning carbon.
- (b) Excessive quantities of insolubles (mainly silica) in zinc concentrate leading to

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- Agglomeration and development of bed pressure resulting in lower throughput in roaster,
- Poor settling during leaching,
- Difficulty in filtration due to silicate - gel formation.

Difficulties experienced in treatment of RDM concentrate at HZL smelters motivated R&D activities to improve quality of concentrates.

*Table 3 : Specifications of Concentrates*

ELEMENT	SMELTER SPECIFICATIONS		ACTUAL (LAST 5 YEAR AVERAGE)
	PYRO/HYDRO METALLURGY	I.S.P	
A. Zinc Concentrate			
Pb	1-2.5%	0.1-2.3%	2.38%
Zn	50-53.5%	48-56%	50%
Cu	0.1-0.4%	0.1-0.8%	0.3%
Fe	6.5-8%	1.86-11%	6.66%
Gr.C.	< 0.5%	0.1-0.8%	1.87%
SiO <sub>2</sub>	< 3%	0.3-4%	4.26%
ISM.	< 4%	-	7.02%
B. Lead Concentrate			
Pb	60-70%	46-65%	43.5%
Zn	<4%	1.12%	5.81%
Cu	0.05-1%	0.01-2.2%	2.29%
Fe	4-7%	1.2-9%	5.24%
Gr.C.	<2%	0.2-3%	7.55%
SiO <sub>2</sub>	2-3%	0.3-5%	9.43%
ISM.	3-5%	-	14.6%

## R & D ACTIVITIES

The mineralogical studies revealed that silica in zinc concentrate and graphitic carbon in lead concentrate is in liberated form. A brief account of R&D activities and alternative beneficiation techniques tested on RDM ore are given below :

*1. Reduction of silica in zinc concentrate*

- (a) Various silica depressants tested at laboratory as well as plant scales include Carboxy Methyl Cellulose (CMC), Dig-depress-DC-100, Formaldehyde, Guar Gum derivative, Amalgam, etc. However satisfactory results were not achieved. Tests were also conducted on regrinding of zinc rougher concentrate before cleaning as well as regrinding of middlings. Although it improved the quality to some extent yet did not meet smelter specifications.
- (b) Column flotation test work conducted in-house on 3" pilot scale flotation column of Digester make at Central Research & Development Laboratory (CRDL) as well as by Regional Research Laboratory (RRL), Bhubaneswar and National Metallurgical Laboratory, Madras, indicated amenability of RDM ore to column flotation technology for reduction of silica in zinc concentrate.
- (c) Further to amenability test work on column flotation technology on pilot scale column at CRDL and through other agencies, M/s Engineers India Ltd. was engaged to scrutinise CRDL test data and scale up of column flotation for plant with the help of a simulator developed by them. Extensive experiment were conducted using 3" pilot flotation columns in cleaning circuit with pilot size mechanical flotation cells for roughing of zinc concentrate this test work was aimed to determine flotation parameter like flotation rate constants and carrying capacity, essential for sizing of industrial flotation column.

*2. Reduction of graphitic carbon in lead concentrate*

- (a) Extensive test work was undertaken with various graphite depressants at CRDL but only Aero-depressants and Nigrosine proved to be marginally effective. In-plant modifications of lead flotation circuit were carried out to incorporate pre-graphite flotation based on laboratory test work. Reverse flotation (depression of galena by dichromate) was also adopted after successful in-plant trials. However, due to heavy metal losses in reverse float, pollution control problem related to dichromate water re-usage/disposal and additional cost involved, both circuits are operated only when extremely adverse ore is received.



- (b) Tests conducted by Cominco Engineering Services Ltd. (CESL), Canada and National Metallurgical Laboratory (NML), Madras indicated feasibility of column flotation technique for two stage cleaning. The gravity separation technique, tried at CRDL on Vanner, Wilfley Table etc., gave encouraging results but were not found effective in fine size ranges.
- (c) Multi-gravity Separator (MGS) Tests conducted by Richard Mozley Ltd., UK, with graphite float on MGS were found encouraging. Hence, tests with lead rougher concentrate containing 12% graphitic carbon were conducted on MGS at RRL, Bhopal. Results indicated that the MGS can effectively reduce graphitic carbon. Assistance was also sought from other R&D Institutions like Indian Bureau of Mines (IBM), Nagpur; Tata Research Development & Design Centre (TRDDC), Pune; Lurgi, Germany; Mineral Resources Development Inc. (Rdi), USA, etc. But the extensive test work conducted by them to find a cost effective solution was not successful. However, coarse grinding from the existing >70% to ~ 65% - 200 mesh, as recommended by TRDDC, was adopted.

Thus, HZL decided to try column flotation technology for silica reduction and MGS for graphitic carbon reduction in the respective concentrates.

## COMMERCIAL INSTALLATIONS

### Flotation Columns

Under S&T scheme in collaboration with Ministry of Mines, EIL, NML and RRL Bhubaneswar, HZL as a nodal agency, a project titled Development of indigenous column flotations technology for industrial application at R.D.Mine was taken up. Basic design and engineering for 2 Nos. 1.5 M dia x 10 Mtr. Height for one 900 TPD zinc cleaner circuit prepared by EIL was got vetted by an expert agency M/s Control Inter National S.A.France. These flotation columns were commissioned in March 1998. Integrated flow sheet of flotation columns in zinc circuit is shown in Fig. 5. As per plant results zinc conc. grade of +54% zinc and silica below 3% without affecting recovery is achieved. With the stabilisation of the system, better metallurgical results are expected.

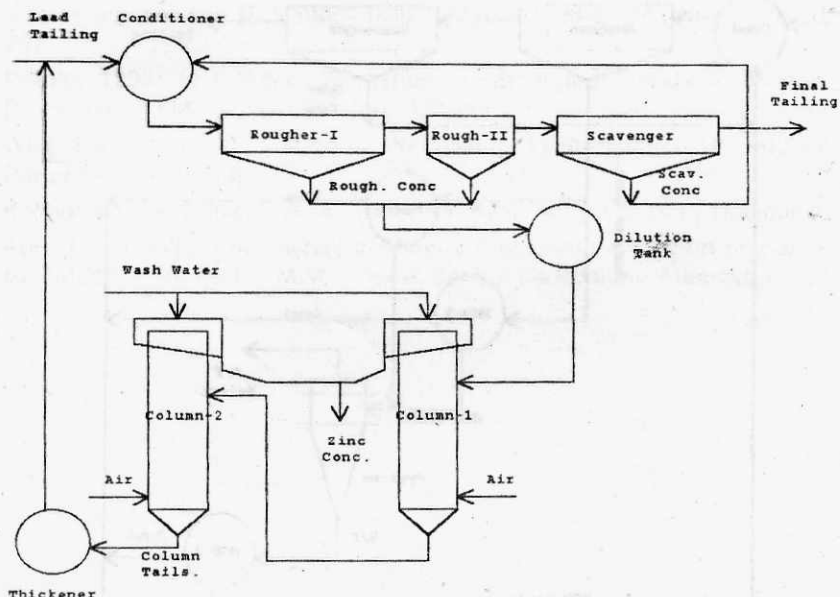


Fig. 5 : Proposed flowsheet with integration of columns in zinc flotation circuit

### Multigravity Separator

Further to extensive R&D work, one MGS unit was installed in the plant on rental cum purchase basis for continuous in-plant testing for a period of four months. A team of scientists from RRL Bhopal was also associated for stabilisation/optimisation of the operating parameters. In-plant trial of MGS circuit gave good lead metallurgy i.e. 45 to 58% Pb. with 2 to 3% gr. Carbon as compared to 40% Pb with 10-15% gr. carbon produced from conventional circuit without MGS.

Based on above in-plant trial, the machine was retained and two more MGS machines with a desliming cyclone unit for one 900 TPD lead circuit were commissioned in June 1998 and are under stabilisation. Integrated lead circuit with MGS is shown in Fig. 6.

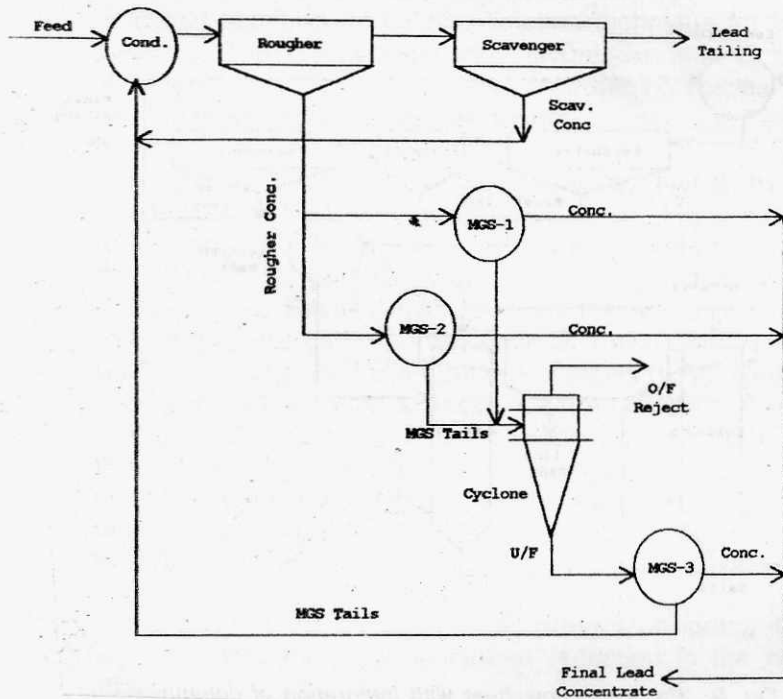


Fig. 6 : Integrated flowsheet of 3 MGS in one 900 t/d lead circuit

Above technologies will be further extended to other two circuits in the plant after stabilisation and optimisation. This will not only improve benefaction plant economic but also smelter efficiencies.

## CONCLUSION

A review of sulphide ore flotation practice, factors affecting the process, innovations and new trends are briefly covered. The developmental efforts, and adoption of new technologies for solving metallurgical problems in HZL's Rajpura Dariba concentrator highlight various practical aspects of sulphide ore beneficiation.

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